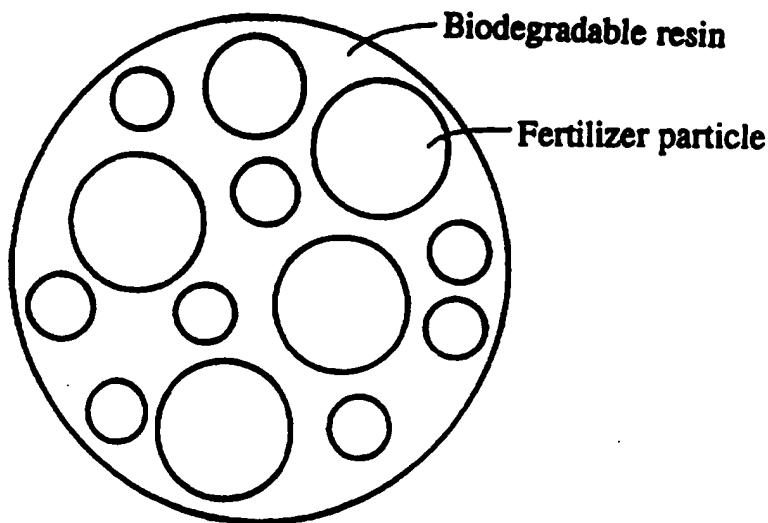




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Yoksam-dong, Kangnam-ku, Seoul 135-080 (KR).(81) Designated States: **CA, CN, JP, US.****Published**
With international search report.(54) Title: **THE CONTROLLED RELEASE MATRIX-TYPE FERTILIZER AND THE PROCESS OF PREPARATION THEREFOR**

(57) Abstract

The invention relates to the novel matrix coating fertilizer, and the process of preparation therefor, wherein the biodegradable coating materials, such as rosin, wax, aliphatic polyesters or the like, are mixed and melted slowly into liquid state, the particles of the fertilizer are added and mixed, the mixed coating materials and the particles of the fertilizer are extruded through the extruder and consequently the particles of the fertilizer are coated with matrix resin. The matrix-type fertilizer of the present invention has reduced the releasing rate of the fertilizer and the prices thereof are very economical since the matrix-type fertilizer of the present invention can be produced by a simple process.

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SPECIFICATION

The Controlled Release Matrix-type Fertilizer and the Process of Preparation Therefor.

5

Technical Field

The present invention relates to the novel matrix-type fertilizer and the process of preparation thereof. In particular, the present invention relates to a matrix-type fertilizer the releasing rate of which is remarkably reduced and a process of preparation, wherein the matrix-type fertilizer ingredient is coated with matrix resin by mixing first resin and fertilizer and then extruding them.

The chemical fertilizers used at present have many problems. On account of high solubility in water, most of the chemical fertilizers have been released into soil very soon after dressing the fertilizer, and this results in low availability of the fertilizer, high dressing of the fertilizer, the contamination of soil due to the accumulation of salts and the waste of labor due to the frequent dressing of the fertilizer. Therefore, the controlled-release fertilizer which can

be consistently released during all the growing period of plants is in a great necessity in order to reduce the loss of the dressed fertilizer and the waste of labor

5

Background Art

There are two kinds of method for preparation of the controlled-releasing fertilizer, i.e., the chemical method and the physical method.

10

In the chemical method, wherein the fertilizer reacts with other substances chemically, the solubility of the fertilizer is reduced by making the fertilizer insoluble salts or less-degradable substances by soil microorganisms.

15

In the physical method, the releasing rate of the fertilizer is reduced by coating the fertilizer ingredient with materials which can prohibit the fertilizer from contacting the water directly.

20

Although several products produced by the chemical method, such as urea-formaldehyde, IBDU(Isobutylidene Diurea), CDU(Crotonylidene Diurea, US Patent No. 3,227,543) or the like, are available, they have some defects that the complicated steps are necessitated for manufacturing and the costs therefor are high.

25

Contrary to the chemical methods, the physical methods, wherein the releasing rate of the fertilizer is controlled by coating the fertilizer particle with the coating materials, have some merits i.e., an
5 excellent sustained effect, easy control of releasing rate and more simple manufacturing processes or the like.

There are several kinds of coating membranes,
10 i.e., semipermeable membrane(thermoplastic resin), impermeable membrane(thermosetting resin), and biodegradable membrane.

The semipermeable membranes are destructed by internal osmotic pressure caused by diffusion and
15 consequently the fertilizer is released. The impermeable membranes have pores through which water penetrates, and consequently the fertilizer is released. The biodegradable membranes are destructed by physicochemical or microbiological activity, and the
20 fertilizer is released.

As the coating materials, synthetic resin and biodegradable materials can be used. Synthetic resins contain thermoplastic resin and thermosetting resin.

Heretofore, the method of coating the fertilizer
25 with coating materials has been employed in order to

make the coated fertilizer, and the representatives available are shown as follows.

Osmocote(US Patent No. 3,233,518) which has been developed by Archer Daniels Midland Company USA, is
5 multilayered with co-polymer of dicyclopentadiene and glycerol ester as a coating material. Although Osmocote has a good quality, there are some defects that costs thereof are high, and it can contaminate the environment due to the difficulty of recovering the
10 used organic solvent.

Sulfur-coated fertilizer(US Patent No. 3,295,950) which has been developed by T.V.A Company USA, is not expensive, but it has defects that it is difficult to control the releasing rate thereof and the soil shall
15 be acidified by the accumulated sulfur.

Besides, a silicate, one of metal oxide, has been used as a coating material, but a silicate tends to be soluble in water easily and the coating materials do not last for a long time (Japanese Patent No. Sho 59-
20 137386).

Thereafter the improved method, wherein the releasing rate is much delayed by recoating the silicate-coated fertilizer with high molecular latex, has been developed(Korean Patent No. 88-153), but it
25 has defects that the costs are high, and that latex is

accumulated in soil.

As mentioned above, since the method of coating the fertilizer with synthetic resin caused the soil-contamination, it has been proposed to develop the method of coating the fertilizer with biodegradable materials instead of synthetic resin.

First, wax has been used as the biodegradable coating materials(US Patent No. 3,232,237), but it has defect that too much wax is necessitated in order to delay the releasing rate sufficiently. Then, the improved method, wherein wax mixed and melted with rosin is used as a coating material, has been proposed(Japanese Patent No. Sho 59-35875).

But in the event that rosin is used as a coating material, there is a severe defect that the pin-holes are generated on the surface of the coating membrane. The reason that the pin-holes are generated is that the forming process of the coating membrane, wherein the coating materials are dissolved and sprayed to the surface of the fertilizer particle, has been conducted at high temperature and the rosin is degraded at high temperature.

In order to prevent the severe pin-hole phenomena generated during the coating process using

biodegradable materials, it has been proposed to use the coating material, which is made by dissolving rosin with organic solvent and then adding the specific substances (Korean Unexamined Publication No. 93-12646).

5 According to the above-mentioned method, as the coating process is performed at low temperature and the generation of pin-holes on the rosin membrane can be reduced, the property of rosin membrane is improved and the releasing rate of the fertilizer is reduced. But
10 in this case, since the softening temperature of coating material is low, the fertilizer particles tend to be coagulated each other and to be worn down during storage and handling.

In order to solve the above-mentioned problems,
15 the method, wherein the fertilizer particle coated with rosin/rosin derivatives, added with high molecular substance and then dissolved in organic solvent is recoated with organic high molecular substance in solution or emulsion solution, has been proposed (Korean
20 Unexamined Publication 94-2203). By this method, the problem generated during handling and storage has been solved, as the mechanical intensity of the coated particles has been increased, but the steps of manufacturing are complicated, and there remains some
25 problems caused by the use of organic solvents.

An object of the present invention is to provide the novel controlled-release matrix-type fertilizer and the process of preparation therefor which is completely different from the process already established.

5 In particular, an object of the present invention is to provide the novel coating process, so called matrix coating process, which is completely different from the previous spraying process, and which has solved the various problems of the previous spraying
10 process.

Disclosure of the Invention

The invention relates to the novel matrix coating
15 fertilizer and the process of preparation therefor, wherein coating materials are mixed and melted slowly into gel state, the particles of the fertilizer are added and mixed, the mixed coating materials and the particles of the fertilizer are extruded through the
20 extruder and consequently the particles of the fertilizer are coated with matrix resin.

The matrix coating process of the present invention is completely different even in concepts from the previous spraying coating process, wherein the
25 coating material is dissolved in solvent, sprayed at

high temperature and coats the particles respectively.

The controlled release matrix-type fertilizer which is coated with matrix resin by the matrix coating process has the merits, such as the followings. The releasing rate can be controlled depending on the weight ratio of matrix resin easily. The contamination of environment can be minimized because the coating material can be biodegradable easily in soil. As organic solvents are not used, so the recovery of solvent is not necessitated. The costs for manufacturing are low and the costs are very economical, as the steps for manufacturing are simple and the process is mass-productive.

Brief Description of the Drawings

Fig. 1 is a drawing for illustrating the cross section of the controlled release matrix-type fertilizer of the present invention coated by the matrix coating process.

Fig. 2 is a drawing for illustrating the cross section of the known fertilizer coated by the spraying coating process.

The present invention will be described in detail

in the following.

The controlled release matrix-type fertilizer contains 5-80 weight % of resin, 20-95% weight % of the fertilizer, and if necessary, 0-20% of drying agent or
5 0-20% of surface compatibilizer can be contained. After mixing and melting these components in the extruder, they should be extruded and coated with resin. All the resin can be used in the process of matrix coating, if they can be melted at 50-120°C i.e.,
10 synthetic resins and biodegradable resins.

In the synthetic resins, there are thermoplastic and thermosetting resin. And in the biodegradable resins, there are rosins such as gum rosin, wood rosin, toloil-rosin, wax and aliphatic polyesters such as
15 polycaprolactone, polylactic acid, esters of diols/diacid or the like. They are used respectively or in combination. Synthetic resins can be prepared by the method of bulk polymerization, suspension polymerization, emulsion polymerization or the like.

20 In the event of using the biodegradable resin, the insoluble metal compounds and polymers which are used in the previous spraying coating process can be added to improve the property of the coating materials. Such an insoluble metal compounds can contain aluminum,
25 calcium, copper, iron, magnesium, zinc fatty acid

compound, aluminum oxide, barium oxide, iron acetate or
silicofluoride compounds of calcium, or lithium, and
they can be used respectively or in combination. High
molecular resin can contain paraffin wax,
5 ethylcellulose or the like.

The fertilizer which can be used for the matrix-
coating process contains urea fertilizer, phosphate
fertilizer, potassium fertilizer, organic fertilizer,
compound fertilizer or the like. These can be used as
10 itself or as grinded pieces of 5-200 μ m particles.

As the surface compatibilizer fatty acids can be
used which can contain oleic acid, stearic acid,
palmitic acid or the like. As the drying agent, calcium
chloride, calcium oxide or the like can be used. In
15 addition to the above-mentioned surface compatibilizer
or the drying agent, any ingredients necessary to the
common coating process can be contained for the matrix
coating process.

20 Another object of the present invention is to
provide a novel process for preparing the controlled
release matrix-type fertilizer efficiently and
economically.

The process of the present invention is
25 characterized the matrix-type fertilizer can be

produced through a single step, wherein all the necessary ingredients are mixed and melted in a extruder, and then extruded.

5 The method extruding can be achieved by mixing through single screw extruder, twin screw extruder, twin screw extruder and then single screw extruder, mixing by Kneader, Banbury or super-mix and then to pass the single screw extruder or twin screw extruder or the like.

10 All the method to prepare the controlled release fertilizer by mixing and melting in the extruder should be within the scope of the present invention.

The present invention which will be described in
15 more detail according to the steps is as follows

Biodegradable resin in the amount of 5-80 weight %, the fertilizer in the amount of 20-95 weight%, the drying agent in the amount of 0-20 weight and the surface compatibilizer in the amount of 0-20 weight %
20 is mixed with super-mix or Banbury and then put into the pre-heated single screw or twin screw extruder.

In the extruder, the fertilizers are grinded and mixed with the other components evenly while the single screw or the twin screw are running at the speed of 50-
25 300rpm. As the resin has lower melting point than the

fertilizer, the fertilizer can be made to particles without melting, although the resins are melted first, therefore the fertilizers are coated with the melted resins and extruded in the coated state with resins.

5 The matrix-type fertilizers are extruded in the form of pellets whose cross section is shown in Fig.1. Fig.1 indicates that the matrix resin contains several particles of the fertilizer. In this state, the fertilizer has shown the same effect as the fertilizer
10 multilayer-coated, and the matrix-type of the present invention has an excellent controlled release effect.

In addition, since the matrix-type fertilizer of the present invention has improved the defect of the spraying coating process that the fertilizer is
15 released suddenly and excessively due to the damage of the coating membrane during transportation or storage, the controlled release property is maintained, even though the coating membrane is damaged.

According to the process of the present invention,
20 the thickness of the coating membrane can be optimized by controlling the amount of the coating materials, 5-80 weight%.

Furthermore, the shape, the size or the like of the fertilizer pellets can be optimized in order not to
25 affect the plants directly by the excessive fertilizer

components. The shape or size of the pellets can be controlled in the range of 1-30mm according to the purpose of fertilization.

It is preferred that inorganic substances, such as talc, kaoline or the like, are sprayed in the amount of 0.1-2 weight% before drying step in order to prevent coagulation between pellets.

The controlled release matrix-type fertilizer of the present invention has solved the problems of the contamination of environment, since no specific additives or no organic solvents are used.

In addition, the scope of available raw materials can be expanded, since any coating material, i.e., biodegradable coating material, such as rosin, wax, aliphatic polyesters used in the previous spraying coating process can be used.

The cost can be decreased and mass production become possible on economical prices, since the process is very simple; the process comprises only mixing step of the raw materials in the super-mixer, Kneader or Banbury and then extruding steps through the extruder, without any pretreatment.

The matrix coating process of the present invention can be employed to various kinds of fertilizers, such as urea fertilizer, phosphate

fertilizer, potassium fertilizer organic fertilizer, or the like.

The fertilizer to which the drying agent is added when the biodegradable resin (rosin, wax, aliphatic polyesters or the mixture) and the fertilizer are mixed and melted diminish the generation of air bubble caused by evaporation during the mixing and fusing step, and consequently the coating membrane becomes so tight that water can not penetrate. This is especially a good property for urea fertilizer since urea fertilizer is very hygroscopic.

In the event that the surface compatibilizer is used, the surface compatibilizer enhances the attraction between coating material and fertilizer.

Due to the good physical properties of the coated fertilizer it is easy to handle during transportation and storage, and easy to control the releasing rate after fertilization.

The mechanical strength of the pellets by the process that materials are extruded through the twin screw extruder or reextruded through the single screw extruder after extruding through the twin screw machine, increases much more than that of the pellets by the process that materials are extruded only through the single screw extruder. The more the mechanical

strength increases, the more the releasing rate becomes low. The fertilizer of high mechanical strength is especially useful to the plants which needs a long-term fertilization.

5

Examples

The following examples will further illustrate the present invention, which by no means limit the present
10 invention.

<Example 1>

This example describes the characteristics of the matrix coating process.

15 5kg of commercially available granular urea fertilizer, 4kg of pulverized gum rosin and 1kg of wax were put into the single screw extruder which was adjusted at 100rpm, 60-100°C. The coated fertilizer by passing through the extruder was cut into pieces having
20 regular sizes. The controlled-releasing test was performed using incubator at 30°C and the amount of the releasing fertilizer was measured. The results were shown in Table 1.

25

Table 1

	1 day	10days	20days	30days	40days
releasing ratio (%)	0.1	5	18	32	53

5

<Example 2>

7kg of commercially available granular urea fertilizer and 3kg of pulverized gum rosin were mixed in super-mixer. The mixture was put into the twin screw extruder, wherein the temperature was maintained at 80-120°C and the speed of the screw was adjusted at 200rpm. The releasing test was performed by the same method as described in Example 1, and the results were shown in Table 2.

15

Table 2

	1 day	10days	20days	30days	40days
releasing ratio (%)	1.0	10	25	40	65

20

<Example 3>

7kg of commercially available granular urea fertilizer, 2.5kg of pulverized gum rosin and 0.5kg of stearic acid were mixed in the super-mix. The mixture

was put into the twin screw extruder, wherein the temperature was maintained at 80-120°C and the speed of the screw was adjusted at 200rpm.

The mixture passed through the twin screw extruder
5 and was put again into the single screw extruder, wherein the temperature was maintained at 60-120°C and the speed of the screw was adjusted at 150rpm. The releasing test was performed by the same method as described in Example 1, and the results were shown in
10 Table 3.

Table 3

	1 day	10days	20days	30days	40days
15 releasing ratio (%)	0.1	5	15	30	50

<Example 4>

0.5kg of oleic acid was dissolved in small amount
20 of acetone, and was mixed with 7kg of commercially available urea fertilizer in the super-mix. 0.5kg of polycaprolactone and 0.5kg of wax were added thereto. The mixture was put into the Kneader preheated to 120°C, mixed and coated by the same method as Example

1. The releasing test was performed by the same method as described in Example 1, and the results were shown in Table 4.

Table 4

5		1 day	10days	20days	30days	40days
	releasing ratio (%)	0.5	7	20	35	60

<Example 5>

10 8kg of commercially available granular urea fertilizer, 1.7kg of gum rosin and 0.3kg of stearic acid were put into the Banbury preheated to 120°C and mixed for 5 minutes, mixed and coated by the same method as Example 1. The releasing test was performed

15 by the same method as described in Example 1 and the results were shown in Table 5.

Table 5

20		1 day	10days	20days	30days	40days
	releasing ratio (%)	0.5	7	20	35	60

CLAIMS

1. The controlled release matrix-type fertilizer characterized by being coated with matrix resin by the process, wherein the composition comprised resin components of 5-80 weight %, fertilizer components of 20-95 weight % is mixed and melted, and then extruded through the extruder.

2. The controlled release matrix-type fertilizer according to Claim 1, wherein the resin components can be melted at the temperature ranging from 50°C to 120°C and thermoplastic resin, thermosetting resin or biodegradable resin can be used respectively or in combination.

3. The controlled release matrix-type fertilizer according to Claim 2, wherein the biodegradable resin can be selected among gum rosin, wood rosin, toloil rosin, wax or aliphatic polyesters such as polycaprolactone, polylactic acid, esters of diol/diacid or the like.

4. The controlled release matrix-type fertilizer according to Claim 1, the fertilizer component can be selected among urea fertilizer, phosphate fertilizer, potassium fertilizer, organic fertilizer or compound fertilizer and can be used as itself or as pulverized

of 5-200 μ m in diameter.

5. The controlled release matrix-type fertilizer according to Claim 1, wherein the surface compatibilizer component of 0-20 weight % and the
5 drying agent component of 0-20 weight % can be added to the resin component and the fertilizer component.

6. The controlled-release matrix-type fertilizer according to Claim 5, wherein the surface compatibilizer can be selected among the fatty acids
10 such as oleic acid, stearic acid, palmitic acid or the like.

7. The controlled-release matrix-type fertilizer according to Claim 5, wherein the drying agent can be selected among calcium chloride, calcium oxide or the
15 like.

8. The process of preparation for the controlled-release matrix-type fertilizer characterized by being coated with matrix resin, wherein the composition comprised the resin component of 5-80 weight %, the
20 fertilizer component of 20-95 weight %, if necessary, the surface compatibilizer of 0-20 weight % and the drying agent of 0-20 weight % are mixed and melted, and then extruded through the extruder.

9. The process according to Claim 8, wherein the
25 extruding method can be selected among the methods; the

method of passing through the single screw extruder or the twin screw extruder, repassing through the single screw or the twin screw extruder after passing the twin screw extruder, passing through the single screw or the
5 twin screw extruder after mixing in Kneader, Banbury or super-mix.

1/1

Fig. 1

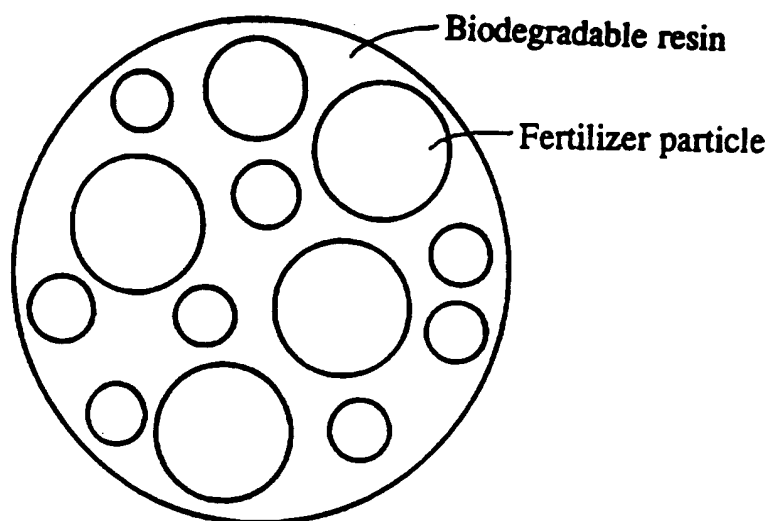
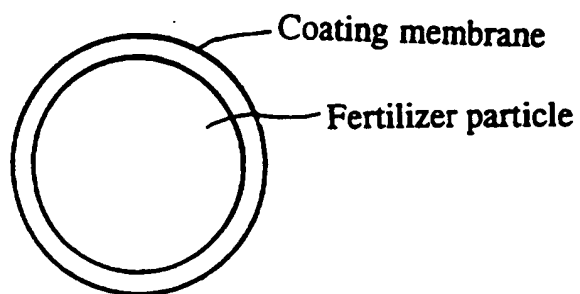


Fig. 2



INTERNATIONAL SEARCH REPORT

International application No.

PCT/KR 95/00159

A. CLASSIFICATION OF SUBJECT MATTER

IPC⁶: C 05 G 5/00, 3/00

According to International Patent Classification (IPC) or to both national classification and IPC

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Minimum documentation searched (classification system followed by classification symbols)

IPC⁶: C 05 G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 93/19 023 A1 (HENKEL KGAA) 30 September 1993 (30.09.93), claims; page 11, line 22 - page 12, line 2.	1-9
X	FR 2 301 497 A (SUMITOMO CHEMICAL CO. et al.) 17 September 1976 (17.09.76), page 7, lines 2-20; claims 25,26.	1,2,4,8
A	EP 0 094 513 A2 (AKZO) 23 November 1983 (23.11.83), claims; page 4, line 18 - page 5, line 29.	1-9
A	US 3 295 950 A (G.M. BLOUIN et al.) 03 January 1967 (03.01.67), claims.	1-9

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT
Information on patent family members

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Le Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche	Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family member(s) Membre(s) de la famille de brevets	Datum der Veröffentlichung Publication date Date de publication
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